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13. ABSTRACT *(Maximum 200 Words)*

Substantial effort was used for designing, constructing and commissioning the "In-situ Atomic Level Growth and Characterization Facility", whose capital equipment was funded by an AFOSR DURIP (AF/F49620-98-1-0341). An ultra-high vacuum sample transfer system and a variable temperature scanning tunneling microscope were attached to two already existing molecular beam epitaxy systems and surface science equipment. In-situ sample transfer between the different systems allowed for better sample fabrication and characterization. The scanning tunneling microscope demonstrated atomic resolution images of gallium arsenide and erbium arsenide single crystal surfaces. A technique for controlling interfacial reactions and phase formation was developed for controlling metal penetration depth and electrical properties of elemental metal contacts on gallium arsenide. One student received a Masters Degree in Materials Science.

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Final Report: **AASERT: Rare Earth Arsenides, Magnetic Semi-Metal Epitaxy for Opto-Electronics (AF/F49620-97-1-0473)**

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Executive Summary

Significant effort was devoted to designing and building an ultrahigh vacuum transfer system which would integrate a variable temperature scanning tunneling microscope and a surface science analysis chamber with two molecular beam epitaxy machines to enable in-situ atomic level studies of rare-earth arsenide epitaxial growth on GaAs surface with the aim to control the formation of rare-earth arsenide/GaAs heterostructures. Effort was also focused on the control of interfacial reactions in metal/semiconductor interfaces together with the use of rare-earth arsenide epitaxial layers as diffusion barriers. By the end of the program, the successful installation and integration of a variable temperature scanning tunneling microscope (VTSTM) and a surface science chamber to two molecular beam epitaxy machines was completed. Atomic resolution scanning tunneling microscopy images of in-situ grown GaAs and rare-earth arsenide surfaces were achieved. The development of techniques for controlling reactions and phase formation at metal/semiconductor contact interfaces and how this can be used to control the electrical properties was also accomplished. Substantial reduction in interfacial reactions was achieved with thin (~5 monolayer thick) rare earth arsenide diffusion barrier layers.

Most Significant Advancements and Conclusions

- The AASERT provided student support for the successful installation and operation of the instrumentation, including a variable temperature scanning tunneling microscope, purchased using the AFOSR DURIP equipment grant: "In-situ Atomic Level Growth and Characterization Facility" (AF/F49620-98-1-0341) for integrating the growth and characterization facilities.
- Routine sample transfer involving 5 different sample mounts between the two MBE machines of different manufacturers, a surface science analysis chamber and the variable temperature scanning tunneling microscope was demonstrated.
- Obtained atomic resolution scanning tunneling microscopy images of epitaxial rare earth arsenide surfaces for the first time.
- Demonstrated a novel technique for controlling interfacial reactions and phase formation to control penetration depth, semiconductor epitaxial regrowth and electrical properties of metal/semiconductor contacts.

People Involved with Research

PI: C.J. Palmstrøm
Postdocs: A.H. Bensaoula
Visiting Scientist: T.G. Finstad
Graduate Students: D. Caldwell, Z. Hilt, A. Grant, L.-C. Chen, B.D. Schultz, J. Farrer, J.W. Dong
Graduate Students Supported: Douglas Caldwell, Zach Hilt, and Andrea Grant.

Theses Granted

Master Thesis:

Douglas A. Caldwell "Regrowth of GaAs Through Controlled Solid Phase Thin Film Reactions", October, 1998

Publications Resulting from this Proposal:

1 D. A. Caldwell, L.-C. Chen, A. H. Bensaoula, J. K. Farrer, C. B. Carter and C. J. Palmstrøm, "In-situ regrowth of GaAs through controlled phase transformations and reactions of thin films on GaAs", Proc. Mat. Res. Soc. Symp., **514**, 455 (1998)

2 D. A. Caldwell, L. C. Chen, A. H. Bensaoula, J. K. Farrer, C. B. Carter and C. J. Palmstrøm, "In-situ controlled reactions and phase formation of thin films on GaAs", J. Vac. Sci. Technol. B, **16**, 2280 (1998)

3 L. C. Chen, D. A. Caldwell, T. G. Finstad and C. J. Palmstrøm, "In-situ formation, reactions, and electrical characterization of MBE-grown metal/semiconductor interfaces", J. Vac. Sci. Technol. A, **17**, 1307 (1999)

4 L. C. Chen, D. A. Caldwell, T. A. Müller, T. G. Finstad, W. Schildgen and C. J. Palmstrøm, "MBE growth and in-situ electrical characterization of metal/semiconductor structures", J. Crystal Growth, **201/202**, 146 (1999)

5 L. C. Chen and C. J. Palmstrøm, "*In-situ* electrical determination of reaction kinetics and interface properties at MBE-grown metal/semiconductor interfaces", J. Vac. Sci. Technol. B, **17**, 1877 (1999)

Table of Content

Installation and commissioning of the "In-situ Atomic Level Growth and Characterization Facility"

This grant provided student support for the design, installation and operation of the equipment funded by an AFOSR DURIP (AF/F49620-98-1-0341) for an "In-situ Atomic Level Growth and Characterization Facility". The existing equipment prior to the implementation of the proposal consisted of three independent systems: a modified VG V80H MBE with Auger electron spectrocopy and low energy electron diffraction systems in its preparation/analysis chamber, a Riber 1000 MBE and a Riber surface science chamber with X-ray photoelectron (XPS) and Auger electron spectroscopies (AES) and a low energy electron diffraction system (LEED). The MBE systems are shown in blue and the surface science chamber in pink in Fig. 1. The DURIP proposal provided funding for the equipment needed for interconnecting these three independent systems (shown in green in Fig. 1) and a new variable temperature scanning probe microscope (VTSTM) (shown in red in Fig. 1). However, the DURIP did not support a student for designing, implementing and operating the equipment. This AASERT provided student support for this project.

Design Issues and Basic System Design

A major concern at the outset was vibration isolation of the VTSTM system. The VG MBE system uses two cryopumps and a turbo pump, all of which produce significant vibration. In addition, air handling and other equipment results in a noisy laboratory both from direct mechanical coupling to the equipment but also form airborne noise. This necessitates the need for careful vibration isolation of the VTSTM and the choice of a VTSTM system which is least susceptible to vibration (i.e has the best vibration isolation).

The different existing systems to be interconnected used different sample manipulation and sample holders. This made their integration particularly challenging. The VG V80H MBE system and the VG V80H chemical beam epitaxy (CBE) system (which will be interconnected at a later date) both use wobble sticks with samples loaded horizontally. The Riber systems use magnetically coupled sample transfer arms with the samples mounted vertically. The Riber 1000 MBE system uses 1.5" diameter Mo blocks, the Riber surface science chamber 2" diameter Mo blocks, the VG MBE system 20 mm x 30 mm Mo blocks with dovetails, the VG CBE system a 3" diameter Mo block and the Omicron VTSTM small 19 mm x 21 mm rectangular blocks with a small tab for a pincer wobble stick transfer.

As both of the VG systems have multiple wobble sticks in their preparation chambers and the Riber systems use only one magnetically coupled loading arm for each system, a transfer system which was compatible with the VG method of transport was considered to be the simplest to implement. A bidirectional transport system was

designed to attach to the end of the VG MBE preparation analysis system for transporting samples from the VG MBE system to the XPS. A second transport system, a VG R2P2 system, which consists of a rotary motion and a rack and pinion drive (Fig. 2) was also designed. This latter transport system allows the sample to be moved out of any of 8 different ports in the R2P2 transfer chamber (Figs. 1 and 2). The sample is loaded on and off the transfer systems using a wobble stick.

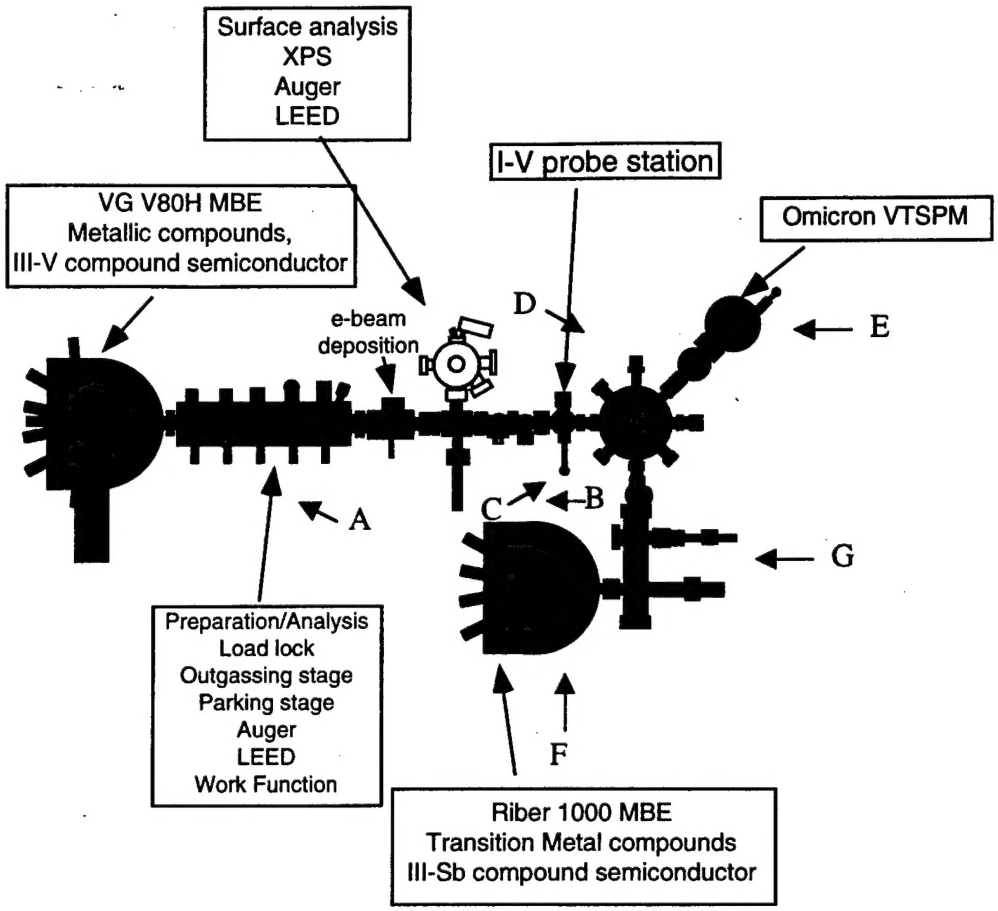


Fig. 1 Schematic of the In-situ Atomic Level Growth and Characterization Facility. The colored capital letters correspond to where the photographs in fig. 3 were taken.

Transfer Chambers and Mechanism

Prior to finalizing the design, a number of critical problems were addressed which included:

- The problem of transferring samples between 5 different sample holders due to different manufacturers' systems (Modified VG V80H with 20 mm x 30 cm MBE molybdenum blocks, VG 3 inch MBE blocks, Riber 1.5 inch and 2 inch MBE sample holders and Omicron 19 mm x 21 mm STM holders).

- The need to change the orientation of the sample in-situ because the VG MBE systems and Omicron STM required the samples to be loaded horizontally while the Riber MBE and surface science chamber required the samples to be loaded vertically.
- Vibration isolation between the VG MBE system with two cryopumps and the STM chamber.
- Potential vibration from other equipment running in the laboratory such as: vacuum rotary pumps, turbo molecular pumps, and air handling equipment.
- The desire to be able to utilize all of the other equipment while the STM is scanning.
- Loading of STM tips and developing tip cleaning procedures.

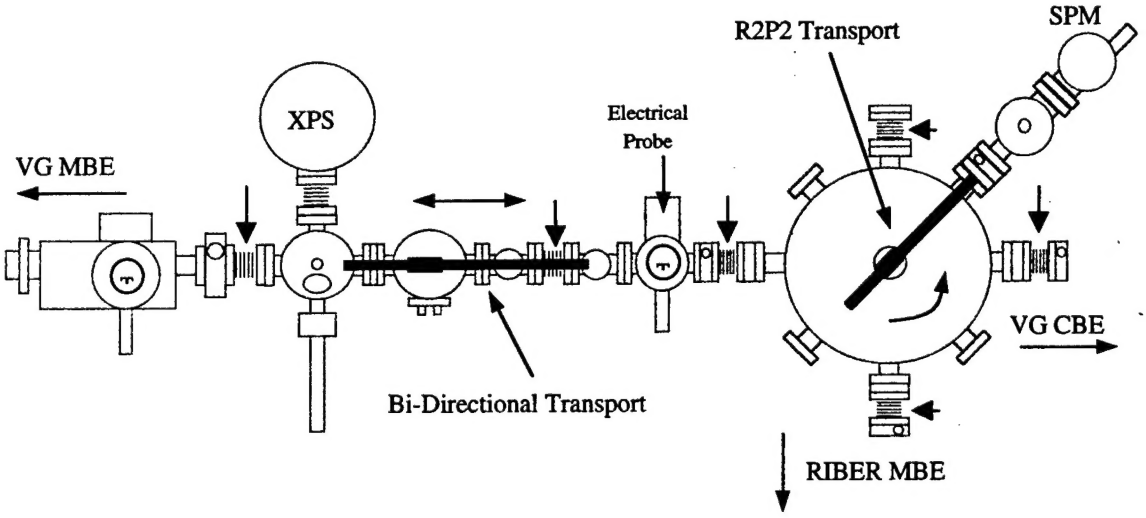
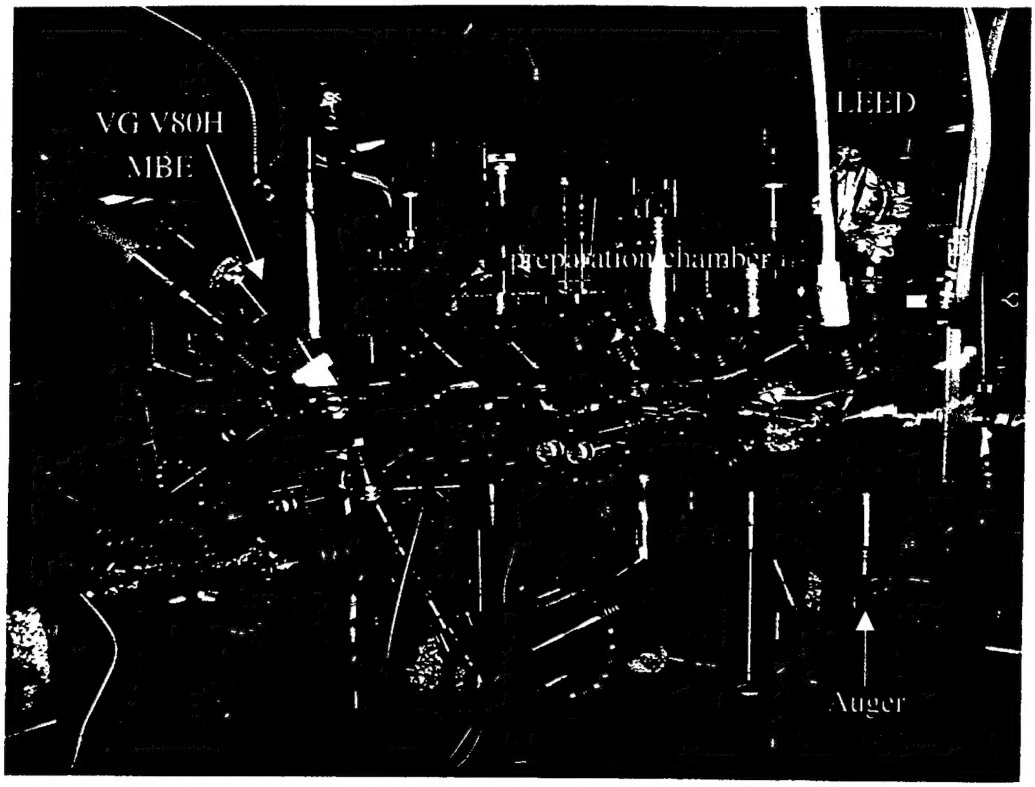
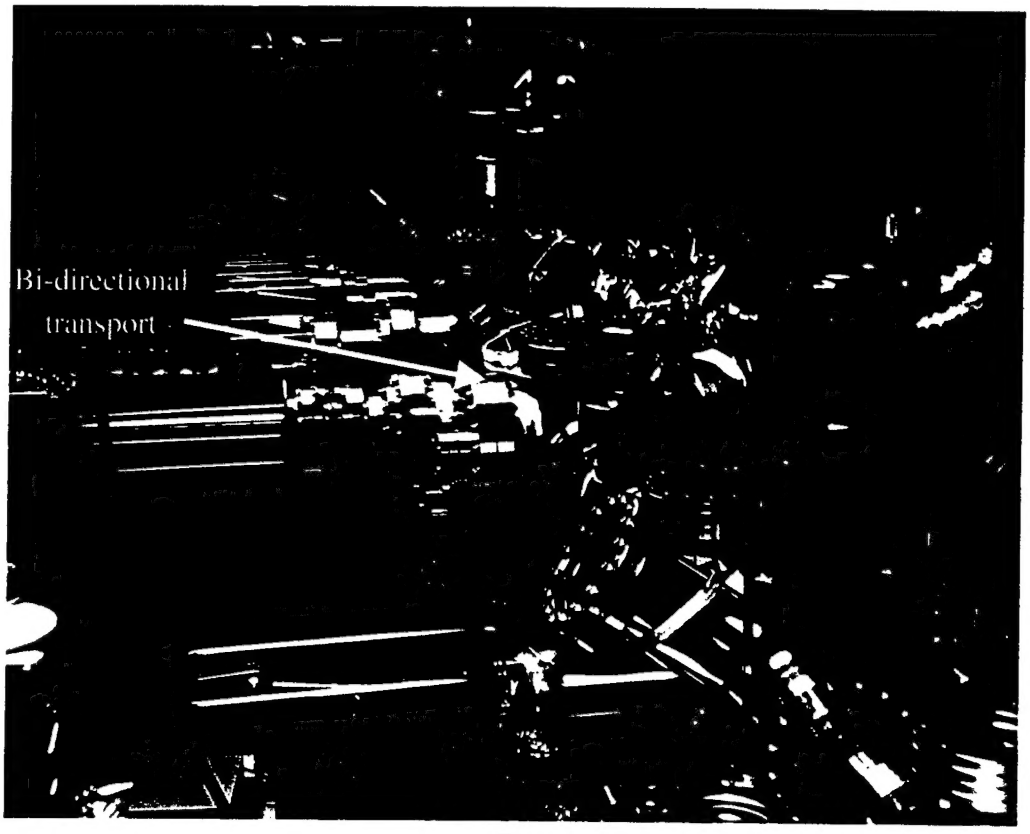


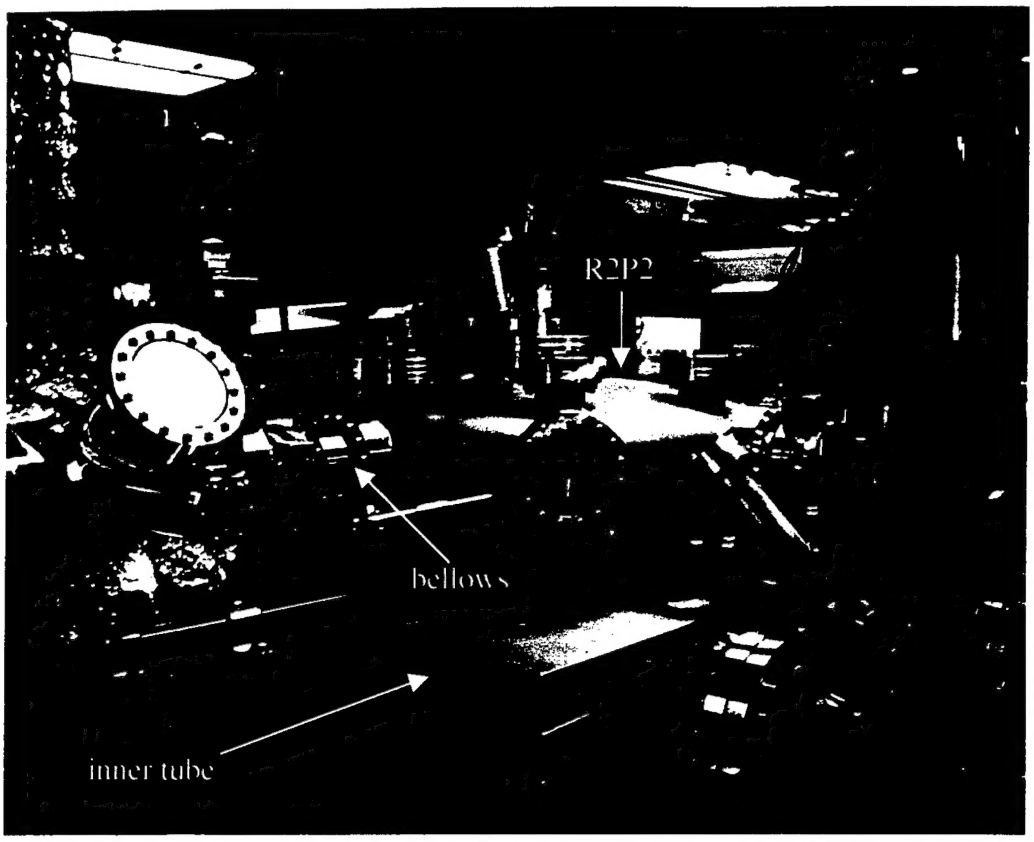
Fig. 2 Schematic of the basic sample transfer mechanism. A bi-direction transport is used to transfer samples from the VG MBE to the second sample transport mechanism, the VG R2P2, and the Riber surface science chamber with XPS. The R2P2 tranport system allows the sample to be extended out of any of the 8 ports on the R2P2 chamber. One port is attached to the VTSPM and another to an additional tranfer chamber to the Riber 1000 MBE system. The VG CBE system will be attached at a later date. The red arrows indicate positions of welded bellows for vibration isolation.


VG V80H
MBE
LEED
preparation chamber
Auger

A


Bi-directional
transport

B



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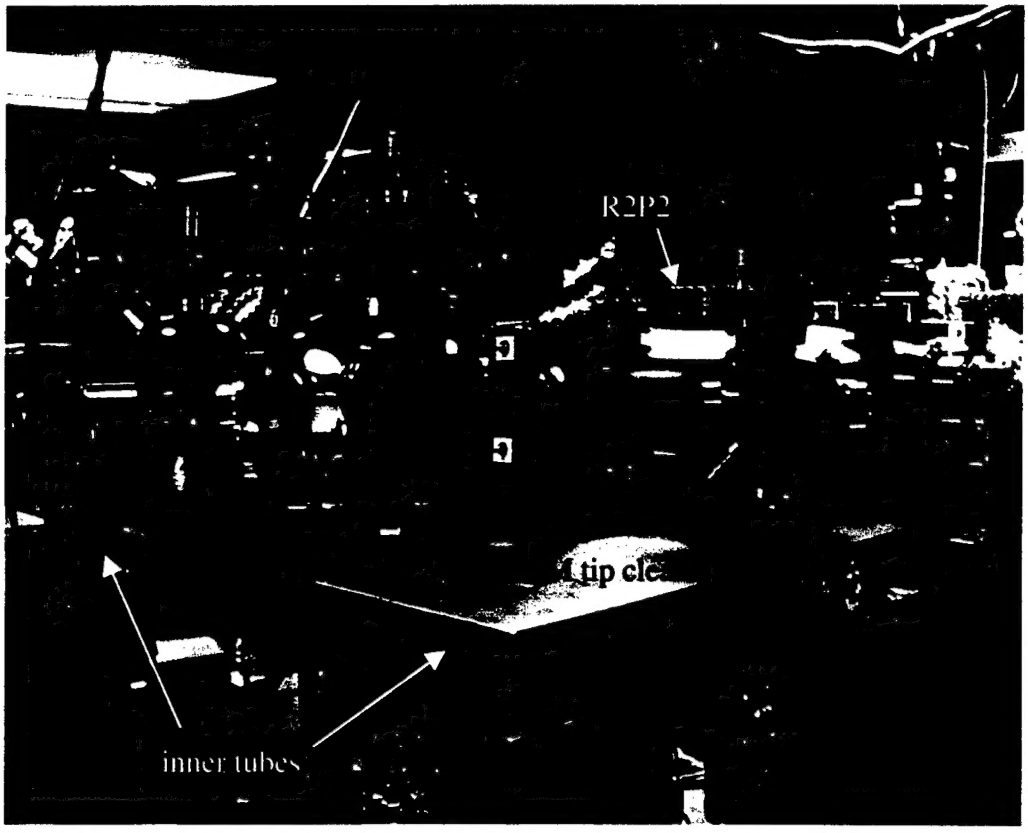

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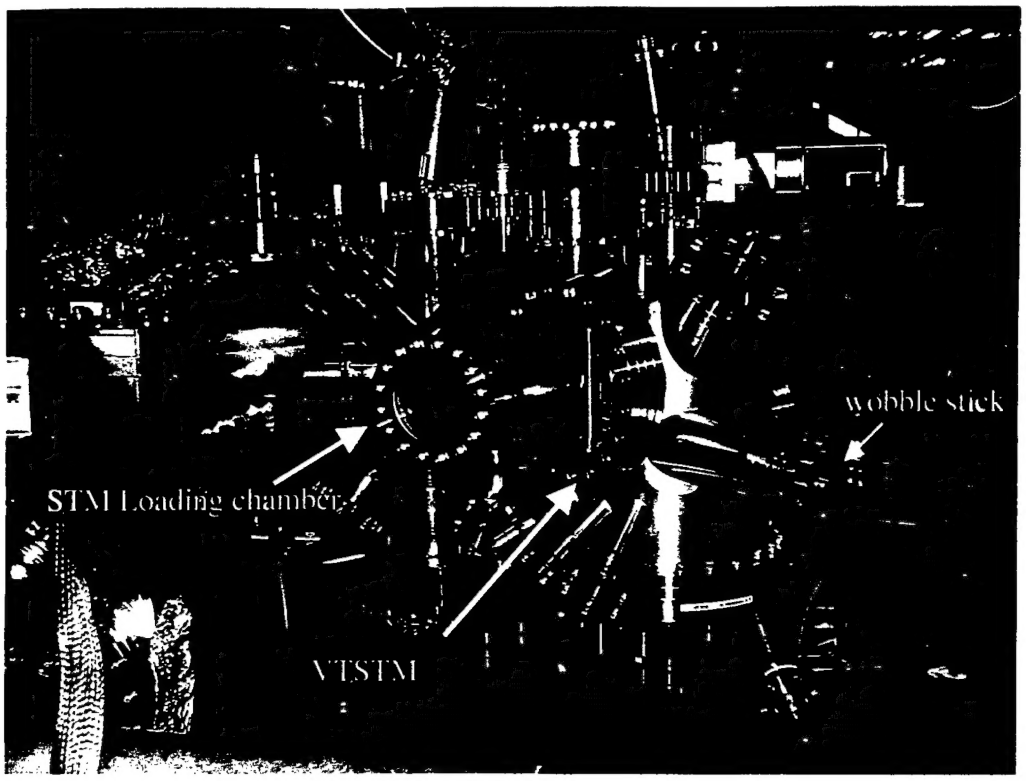

E

F

G

Fig.3 Pictures taken of the In-situ Atomic Level Growth and Characterization Facility. They were taken at the positions labeled in Fig. 1 with the same red letters.

Sample Block Design and Flipping from Horizontal to Vertical Sample Position

The approach taken was to standardize to one primary block that can be transferred from system to system. As the VG MBE and CBE systems and preparation chambers rely on using multiple wobble sticks and a sample transfer trolley to move the sample around, the decision was to use a sample block that was compatible with the wobble stick manipulation. The modified VG V80H with 20 mm x 30 mm MBE molybdenum blocks were small enough that they could be mounted on top of Riber 1.5" and 2" diameter sample holders. In order for this to be possible, the smaller VG sample blocks have to be flipped from being horizontal (VG sample loading) to vertical (Riber sample loading). This was achieved by designing a sample trolley that could be flipped up using a vertically mounted wobble stick. The wobble stick was also designed to lift the sample block out of the trolley holder and into the Riber sample blocks. Figure 4 shows the bi-directional sample transfer trolley in the horizontal and vertical positions at the position for loading into the Riber surface science chamber. The end of the double actiing wobble stick is seen at the top of the pictures. The sample block can be flipped vertically by using the wobble stick to pivot the trolley cart about a hinge (Fig. 4). The sample Mo block inserts into the horseshoe type holder on the transfer trolley. Dovetails on the side of the blocks are used to keep the VG sample holder in place. The holder on the trolley

has been designed such that its center of gravity makes both the vertical and horizontal positions stable. At the bottom of the picture, the end of the Riber magnetically coupled transfer arm can be seen with a 2 inch diameter Riber holder. The front of the sample holder has an additional Mo piece added with the dovetail and a spring clip required to accept the VG holder and keep it from falling out. The VG sample holder in Fig. 4 has an Omicron STM plate mounted on it. In the horizontal position (Fig. 4(a)), the back of the STM plate is visible. The hole in the VG holder allows for direct radiative heating of the STM plate from the back. The front of the holders and the Ta spring clips used to hold the STM plate on the VG holder can be seen when the sample block is flipped to a vertical position (Fig. 4(b)).

A similar sample transfer system was added to the end of the R2P2 rack and pinion transfer arm. This is shown in Fig. 5. In this figure, a sample block that has been modified to accept an Omicron STM plate is shown in the horse-shoe holder. Ta spring clips on the side of the VG holder are used to hold the Omicron STM plate on. The wobble stick that is used to load the sample from the bi-directional onto the R2P2 transfer is visible at the bottom of the pictures. This wobble stick is also used to transfer the sample into the electrical probe station seen in the background. The STM plate is slid off the back of the holder in the STM chamber in order to insert it into the STM. The door that is down when the sample holder is horizontal stops the VG sample block from being pushed off the R2P2 sample holder while inserting the STM plate back on the VG holder in the VTSTM loading chamber.

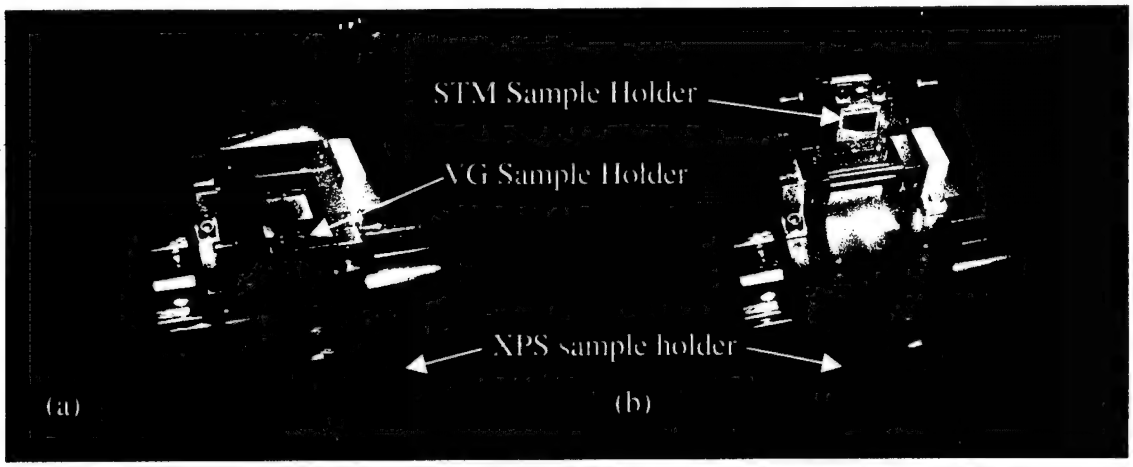

Fig. 4 The bi-directional sample transfer cart. (a) in the horizontal position and (b) flipped into the vertical position using the wobble stick, whose end can be seen to come in from the top. The dove tails for mounting the on the Riber 2" holder for the XPS can be seen in the foreground.

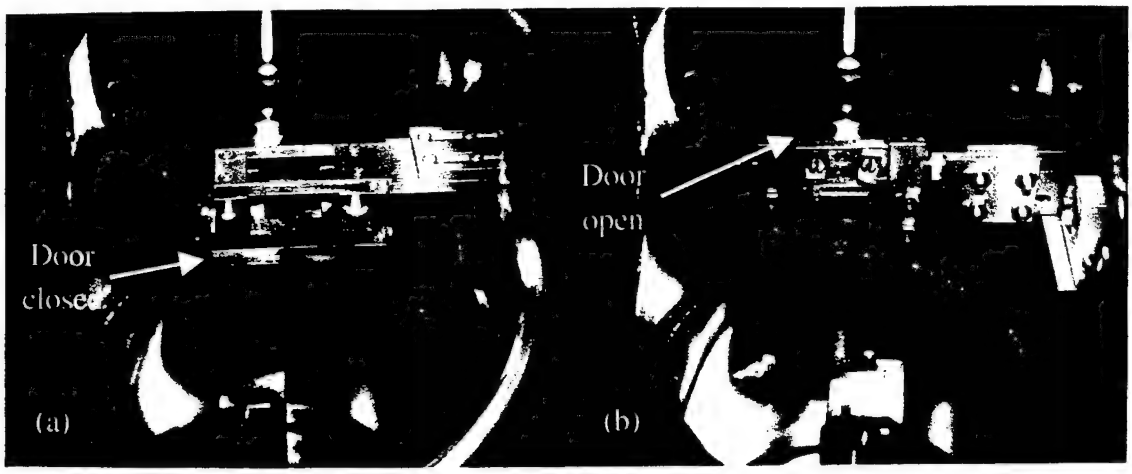

Fig. 5 The end of the R2P2 transfer arm in the position where the sample can be transferred to the bi-directional trolley. (a) shows the sample in the horizontal position and (b) in the vertical. The sample block has a hole in it for direct radiant heating of a SPM sample block when mounted on this VG sample block.

Addition of the VTSTM and Vibration Isolation

A serious concern at the outset of this project was vibration isolation. The modified VG V80H MBE system has two cryopumps on it. One is located at the far end of the growth chamber and the other in a small e-gun deposition chamber between the preparation chamber and the bidirection transport chamber (see Fig. 1). Cryopumps produce significant low frequency vibrations. In order to decouple the VTSTM from the cryopumps, three sections of welded bellows were used between the R2P2 and the VG V80H MBE system (see Figs. 2 and 3). Fig. 2 shows the 4 bellows that are attached to the R2P2. By use of spreaders, which are not coupled to the R2P2, the bellows are expanded such that there is no lateral force on the R2P2. The VTSTM and the R2P2 are mounted on a 1 inch thick Al plate, which is supported on four inner tubes (see Figs. 3 C and D). This allows the R2P2 and VTSTM to be decoupled from the rest of the system and the floor. The cryopump compressors are also supported on inner tubes.

The VTSTM loading chamber is mounted between the R2P2 and the VTSTM chamber itself. The loading chamber serves a number of functions. One is for the unloading of STM plates from the VG sample plates. The VTSTM wobble stick (Figs. 2 and 3E) reaches into the loading chamber and unloads the STM plate while the VG sample holder remains on the R2P2 loading arm. The loading chamber also has a 600 l/s ion pump used to pump the VTSTM. A rotary manipulator arm has an additional sample heater mounted. This allows annealing of samples to occur without the need to transport them back to an MBE system.

In order to minimize electrical noise, the R2P2 and VTSTM are electrically isolated from the rest of the system. This is achieved by using specially designed ceramic breaks in the vacuum chambers between the R2P2 and other systems.

System Performance

Initial studies were performed to determine the optimum annealing conditions post GaAs growth for the formation of the smoothest surfaces with minimal adsorbed GaAs clusters. Fig. 6 shows STM images of GaAs(100) As-rich (2x4)/c(2x8) surfaces with similar RHEED and LEED patterns. Figure 6(a) shows the typical surface after GaAs(100) growth at 580°C followed by a post growth anneal for 15 min at the same temperature with an As_4 flux Although the As dimer rows can easily be seen, there are large numbers of small second layer clusters on the surface. A two step post growth anneal with reduced As_4 fluxes at 530°C and at 350°C results in a significant reduction in the small island density and much smoother surfaces (Fig. 6(b)). This procedure was important to develop to enable further studies. Note further that the bilayer steps which run parallel to the As-dimer rows are very straight, whereas the ones running perpendicular to the dimer rows are always much more jagged and consist of many small sections of steps running parallel to the dimer rows, as can be seen in Fig. 6(b).

Figure 7 shows an STM image of the GaAs(100) As-rich (2x4)/c(2x8) surface after 0.04 monolayers of FeCo deposition. This image clearly shows the As-dimers and dimer rows. In the blowup of the central part of the image, the dimer rows can clearly be seen to consist of two dimers and two missing dimers when going perpendicular to the dimer rows and as clusters of two dimers (four As atoms) with a small gap to the next when going along the dimer row. This would be consistent with the (2x4)/c(2x8)β2 or the (2x4)/c(2x8)α surface reconstruction of GaAs(100).

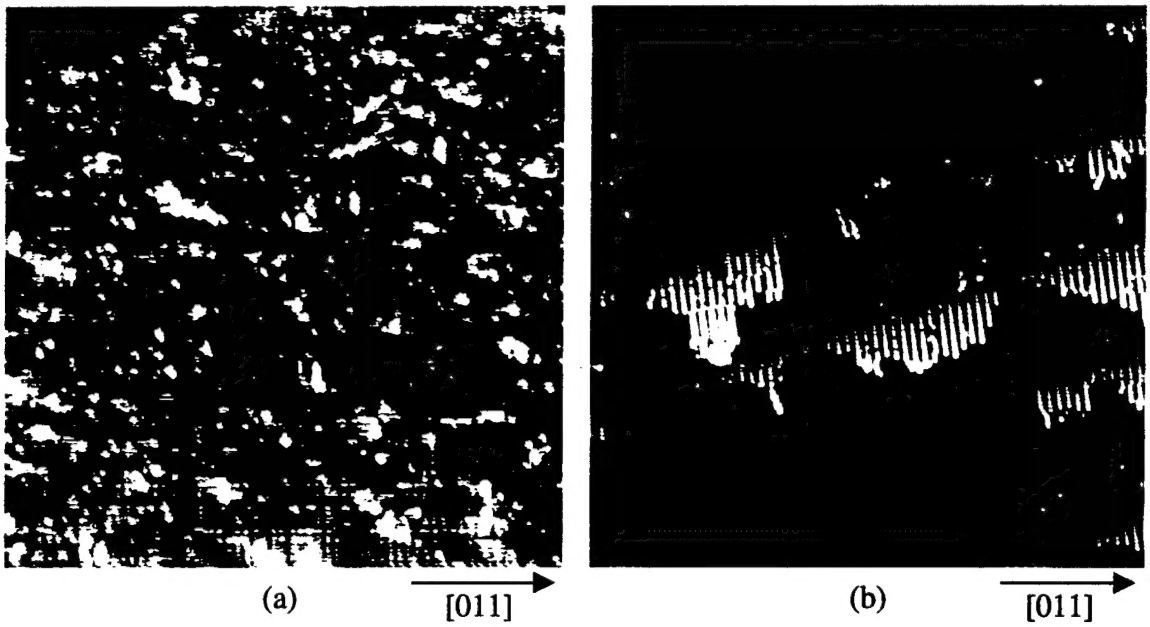

Fig. 6 STM images of GaAs(100) As-rich (2x4)/c(2x8) surface: (a) post growth anneal at 580°C under an As_4-flux and (b) two step post growth anneal under a reduced As_4-flux.

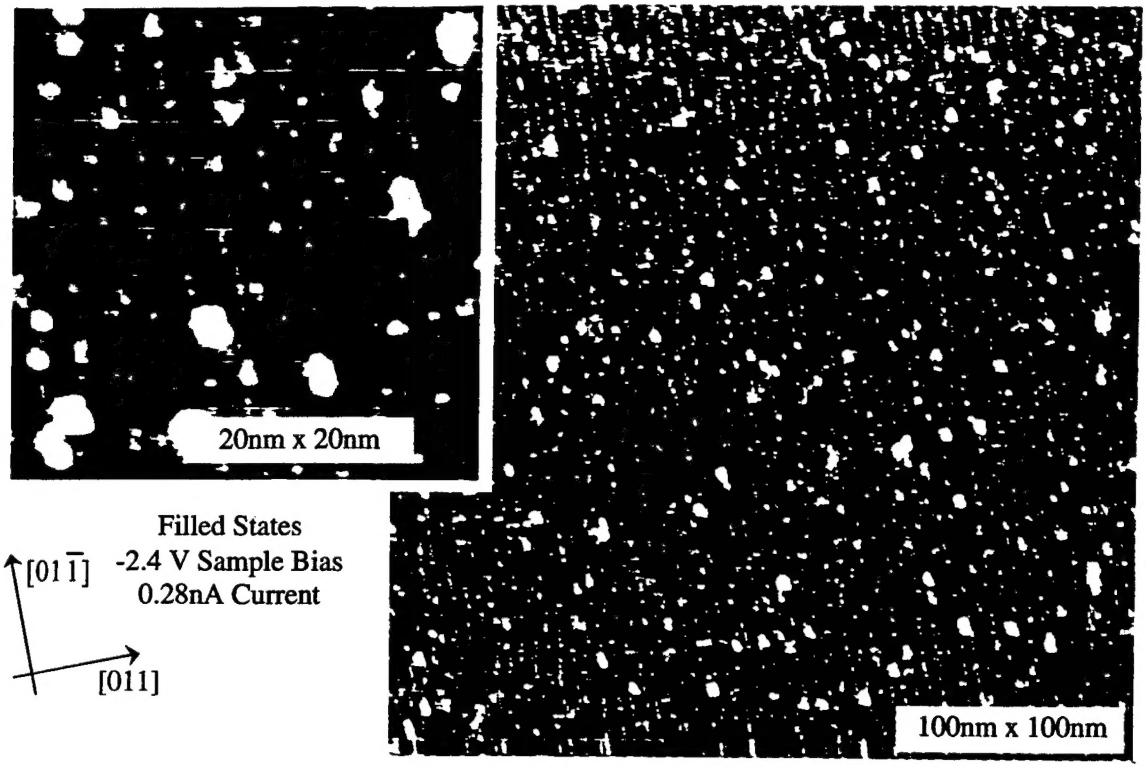

Fig. 7 STM image of GaAs(100) As-rich (2x4)/c(2x8) surface with 0.04 monolayers of FeCo deposition. The image on the top left is a blowup of the central region of the larger image. The As-dimers and dimer rows are clearly visible. The bright clusters are FeCo

Studies of surface structures of $Sc_{1-x}Er_xAs$ grown on GaAs(100) turned out to be even more challenging. Although large scale images were relatively straightforward to obtain, atomic resolution images were much more difficult. This could partially be due to its semi-metallic properties and also the fact that its NaCl crystal structure is not expected to have a surface reconstruction. Fig. 8 shows an STM image of ErAs after a 580°C post growth anneal. The ErAs grows in a monolayer by monolayer fashion. In this image, the ErAs islands have a thickness of one atomic layer. In the figure only three different levels of atomic layers can be seen. A surface corrugation, ~0.3Å in roughness, is visible in the flat regions of the surface. The cause of the corrugation is not known. However, they may be responsible for the weak (1x3)/(3x1) LEED pattern observed after the annealing.

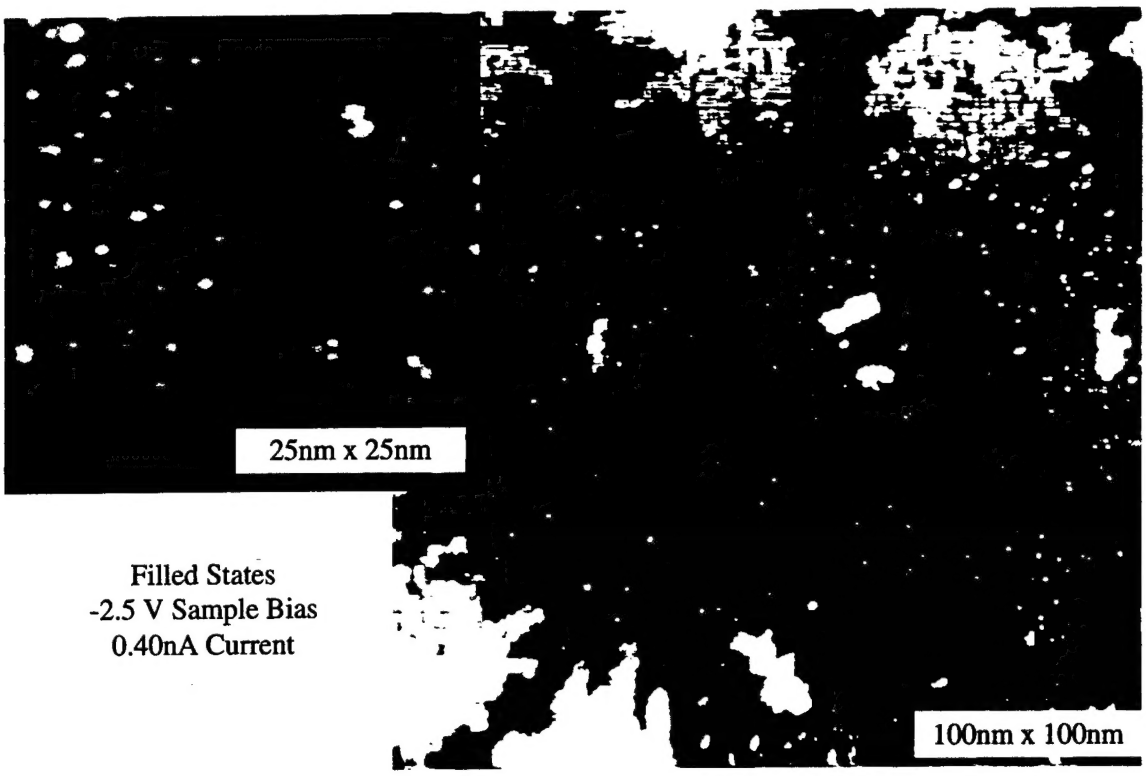

Fig. 8 STM images of from a 6 monolayer thick epitaxial ErAs layer grown on GaAs(100) after a 580°C post growth anneal. A surface corrugation ~0.3Å is evident on the ErAs surface.

All of these STM images were taken with all of the equipment in the laboratory running, including the cryopumps. The image quality remained the same even if the MBE machines were being used to grow heterostructures, resulting in shutter operations. To date, the STM appears to be able to give atomic resolution images while all of the other equipment is being used by other researchers. This makes the system very productive Jumping on the floor next to the STM did have an effect. In general, the main concerns are vibrations of the floor next to the STM and possible acoustic pickup of laboratory background noise. The laboratory has significant airborne noise from the cryopump compressors, air handling, turbo pumps and rotary pumps. However, the noise problems do not appear to be a limiting factor in achieving atomic resolution.

The final conclusion is that the interconnection and VTSTM is working as well or better than expected. Design of new sample holders is required for the implementation of variable temperature STM measurements.

MBE growth and in-situ electrical characterization of metal/semiconductor heterostructures.

Unique techniques were developed as a result of this AASERT on controlling and utilizing interfacial reactions and phase formation to control penetration depth and

electrical properties of metal/GaAs contacts. The results of these studies are summarized in the five publications given in the Executive Summary.